53344

Challenges and Opportunities in Design, Fabrication, and Testing of High Temperature Joints in Ceramics and Ceramic Composites

M. Singh
Dynacs Engineering Co.
NASA Glenn Research Center
Cleveland, OH 44135 (USA)

Abstract

Ceramic joining has been recognized as an enabling technology for successful utilization of advanced ceramics and composite materials. A number of joint design and testing issues have been discussed for ceramic joints in silicon carbide-based ceramics and fiber-reinforced composites. These joints have been fabricated using an affordable, robust ceramic joining technology (ARCJoinT). The microstructure and good high temperature mechanical capability (compressive and flexural strengths) of ceramic joints in silicon carbide-based ceramics and composite materials are reported.

INTRODUCTION

Advanced ceramics and fiber reinforced composite materials are either currently being used, or are under active consideration for use in a wide variety of high temperature applications within the aerospace, nuclear, energy, and process industries. The potential applications of these materials in the aerospace industry include combustor liners, exhaust nozzles, and a number of other aircraft gas turbine and space propulsion components. The land-based applications of these materials include radiant burners, hot gas filters, high-pressure heat exchanger tubes, and combustor liners in industrial gas turbine engines. In addition, there are a number of potential uses of silicon carbide-based materials for the first wall and blanket components of nuclear reactors. At present, the majority of the approaches for the manufacturing of these components yield single pieces. The engineering design and manufacturing of components and systems have become more complicated. As a result, there is an ever-increasing demand for the manufacturing of large and complex shaped parts, which are either quite expensive or very difficult to fabricate. Given these constraints, the small structures could be assembled into large components or structures with a reliable joining or attachment technology. Thus, joining has been recognized as one of the enabling technologies for successful utilization of silicon carbide-based ceramics and fiber reinforced composites in high temperature applications [1-6]. However, the joints must retain their structural integrity at high temperatures and must have mechanical strength and environmental stability comparable to the bulk materials. In addition, the joining technique should be robust, practical, and reliable. It is also interesting to note that high performance and high temperature property requirements for ceramics and fiber reinforced composites and their manufacturability are typically considered independently at the moment. However, these issues have to be addressed simultaneously in the future for the successful implementation of these materials.

In this paper, critical joint design and testing issues are discussed along with the affordable, robust ceramic joining technology (ARCJoinT). ARCJoinT, which is based on the reaction forming approach, is unique in terms of producing joints with tailorable microstructures. The formation of joints by this approach is attractive since the thermomechanical properties of the joint interlayer can be tailored to be very close to those of the base materials. Thermomechanical properties of reaction formed joints in reaction bonded silicon carbide ceramics and fiber-reinforced ceramic composites are discussed.

JOINT DESIGN AND TESTING ISSUES

Numerous joint design and testing activities in the past have been related to metal-metal and ceramic-metal systems. The design accommodates a number of factors including stresses and stress distribution in the joint regions, which are dependent upon joint configuration and chemical and thermal properties mismatch between the joint and substrate materials. For ceramic-metal systems, various joint designs and design criteria have been established [1-3]. A wide variety of testing methods [1-3] have been used to determine the tensile strength, peel strength, flexural strength, shear strength, and compressive strength of the ceramic-metal joints. Unlike the joining technology for ceramic-metal systems, joint design and testing is not well developed and understood for ceramic-ceramic systems since most of the systems are

relatively new. However, there has been some interest in this area in recent years [3-4, 7-17].

In monolithic silicon carbide-based ceramics, compression and flexure tests can provide quite useful information for realistic joint configurations. These tests are not very useful for the evaluation of joint properties in fiber reinforced composites, due to thermomechanical property anisotropy and inhomogeneity coupled with property mismatch with monolithic ceramic joints. In addition, some critical issues associated with the time dependent thermomechanical behavior of joints and their impact on the joint design process have to be addressed.

Recently, a number of critical issues have been raised [17] regarding using the joint strength data for the design of ceramic components. Typically, most of the property data available for design is collected on virgin coupon samples. There are a number of issues raised by designers concerning using this type of data, and some of them are listed below:

- Are joint properties in the component equivalent to coupon properties?
- What is the effect of thermal cycling (number and conditions) on the thermomechanical properties of joints, and is the coefficient of thermal expansion (CTE) independent of number of thermal cycles?
- Are the initial joint properties retained during the part's lifetime?
- Does the material lose modulus and does this change the CTE? If it does lose modulus, can we use this to know when to remove the part?
- Do the oxidation resistance and damping (acoustic signature) change?

If the ceramic and composite materials have to be used at high temperatures under extreme operating conditions, these property issues have to be considered in the joint design process. Another critical design and testing issue is the determination of the stress-state at the joint, namely, tensile, shear, or a combination of tensile and shear stresses under operating conditions. The design of joints must also take into account the response of joints to temperature changes. There is a strong need for the determination of time dependent thermomechanical properties of the joints in ceramics and composites [17].

EXPERIMENTAL PROCEDURES

The REFEL™ Reaction Bonded-SiC materials used in this study were obtained from Pure Carbon Co., St. Marys, PA. These materials were fabricated by the reaction bonding of coarse and fine silicon carbide grains with silicon using a liquid silicon infiltration process. As-processed samples were sectioned, mounted, and polished for metallographic studies. For joining studies, silicon carbide pieces were machined from SiC plates. These pieces were cleaned in acetone and dried before joining.

The SiC (Hi-Nicalon™) fiber reinforced /Melt Infiltrated SiC (MI-SiC) matrix composites used in this study were fabricated at NASA Glenn Research Center, Cleveland, OH. These composites were fabricated with 8-HS weave SiC (Hi-Nicalon™) fiber with ~0.5 μm BN and ~3 μm thick silicon carbide interface. The SiC matrix was fabricated via a reactive melt infiltration process. For the preparation of specimens for mechanical testing, composite bars of 4" x 1" x 0.1" dimensions were used. The composite specimens were aligned to form butt joints. As-fabricated surfaces were cleaned in acetone and dried before joining.

A flow diagram of the affordable, robust ceramic joining technology (ARCJoinT) is given in Figure 1 [9, 12]. The joining process begins with the application of a carbonaceous mixture to the joint area, holding the items to be joined in a fixture, and curing at 110-120°C for 10 to 20 minutes. This step fastens the pieces together. Then, silicon or a silicon-alloy in tape, paste, or slurry form is applied around the joint region and heated to 1250-1425°C (depending on the type of infiltrant) for 10-15 minutes. The molten silicon or silicon- refractory metal alloy reacts with carbon to form silicon carbide with controllable amounts of silicon and other phases as determined by the alloy composition. Joint thickness can be readily controlled in this process, by controlling the properties of the carbonaceous paste and applied fixturing force.

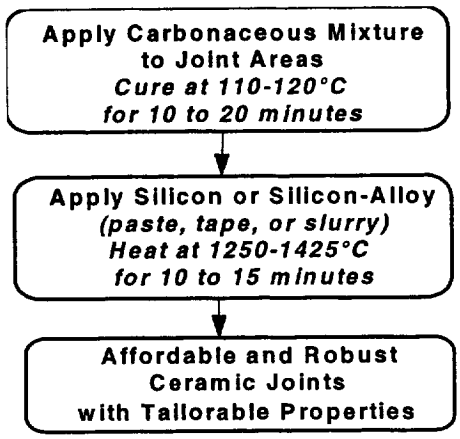


Fig. 1: Schematic of the ARCJoinT joining process for silicon carbide-based ceramics and fiber reinforced composites.

A wide variety of silicon carbide-based ceramics and ceramic matrix composites, consisting of different sizes and shapes, have been joined using this technology (Figure 2). Microstructural characterization and mechanical properties of joints in a number of ceramic and composite systems have been reported in other publications [7-17]. After joining, the microstructure and mechanical properties of joints were characterized at different temperatures.

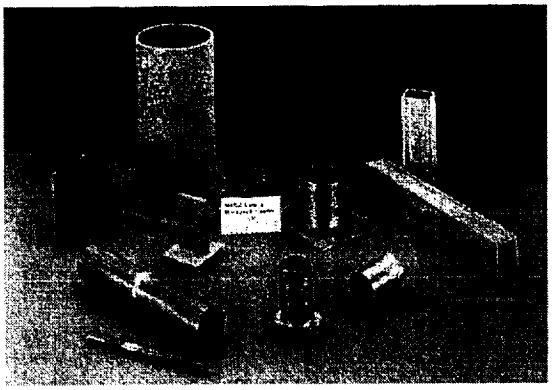


Fig. 2: Photograph showing components fabricated by joining silicon carbide ceramics and fiber reinforced silicon carbide matrix composite subelements.

High temperature compressive strength of joints in reaction bonded silicon carbide (RB-SiC) was measured. Small specimens of 2.3 x 2.3 x 4 mm size were machined from the large joined specimen. In these samples the joint was at a 45° angle with the compression (longer) axis. The specimens were tested in compression at a constant strain rate of 2×10^{-5} s^{-1} for temperatures ranging from 1235°C to 1420°C, in air. Testing was performed using a screw driven Instron universal testing machine model 1185 with a furnace mounted on its frame. Alumina rods with SiC pads were used. The load/time behavior was monitored on a chart recorder. Details of the testing procedure are given in another publication [15-16].

Mechanical properties of joints in fiber reinforced composites were evaluated in flexure mode. Flexure test specimens were machined from the joined bars, with joints centrally located. Four-point flexural strength testing was carried out using the MIL-STD-1942 (MR) configuration B specimens with 20 mm inner and 40 mm outer spans. Flexure tests were conducted at 25, 800, 1200, and 1350°C in air. Three specimens were tested at each temperature. After testing, fracture surfaces were examined by optical and scanning electron microscopy to identify the failure origins.

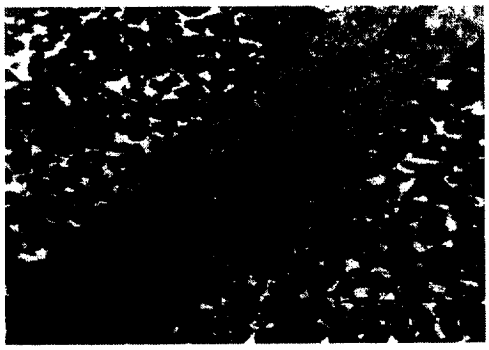
RESULTS AND DISCUSSION

Monolithic Silicon Carbide (RB-SiC):

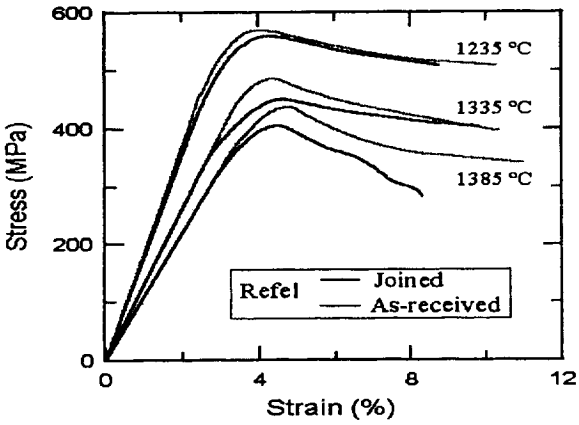
The microstructure of the as-received RB-SiC is composed of very large α-SiC grains (with sizes up to ~ 30 μm) and considerably smaller grains (with sizes ranging from 1-5 μm), and ~ 12 ± 2 % volume fraction of silicon. The typical microstructure of the joints is shown in Figure 3 (a). The white and gray areas are Si and SiC, respectively. The silicon distribution in the joint area was clearly much finer than the bulk RB-SiC. The joint thickness for the RB-SiC was 58.2 ± 1.2 μm. The volume fraction of silicon in the joint was 8 ± 2.2 %. Detailed analyses of RB-SiC base material and the joints have been described elsewhere [15, 16].

The stress-strain curves obtained during the constant strain rate compressive experiments are shown in Figure 3 (b). The joints of RB-SiC can undergo significant plastic deformation (the total strain is over 10% without failure). The strength of the joints gradually decreases with temperature. Typically, the strength of the joints in RB-SiC is about the same as that of the bulk material. The joints of RB-SiC deformed uniformly at 1235°C and 1335°C with the fracture plane often being perpendicular to the joint plane. At the highest testing temperature used for RB-SiC (1385°C), silicon was observed on the sample surface and the deformation in the joint occurred mainly by shear. There was extensive presence of silica on the samples deformed at 1385°C.

The compressive behavior of the joined SiC depends on the relative strength of the joint and the material joined, the interphase between the joint and the bulk, and the microstructure and phase distribution of the joint. In this case, the joint and the bulk material have a similar type of fabrication process in which some silicon remains as the intergranular phase (RB-SiC having larger grains and larger amount of silicon). Due to this similarity the joined sample deforms homogeneously. The failure does not occur along the joint and the joint is as strong as bulk RB-SiC. The low amount of silicon in the joint, its crystallinity, and the interconnectivity of the SiC phase are key factors in achieving good joint strengths.



(a)



(b)

Fig. 3: (a) Joint microstructure and (b) stress-strain behavior of joints under compression at different temperatures.

Fiber Reinforced Composites

A scanning electron micrograph of a fracture surface of a reaction formed joint-MI SiC/SiC composite interface is shown in Figure 4. The joint thickness is ~125 μm. Detailed microstructural examination indicated a small amount of porosity was present in the joint. There is no visually detectable damage to the fibers.

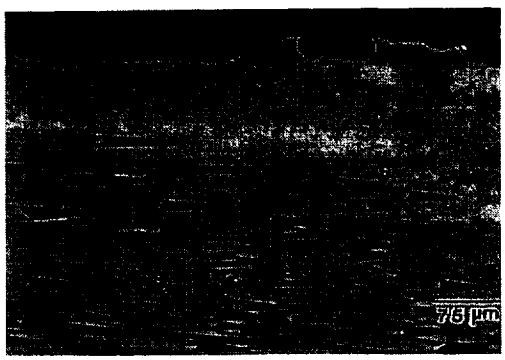


Fig. 4: Microstructure of reaction formed joint-MI SiC/SiC interface.

A summary of flexural strengths of the reaction formed joints in MI SiC/SiC composite materials is shown in Figure 5. The average four point flexural strengths of joined specimens at 25, 800, 1200, and 1350°C were 99±3, 112±3, 157±9, and 113±5 MPa, respectively. The average four point flexural strength of the as-fabricated composites is 360 MPa at room temperature. In the joined MI SiC/SiC materials, fracture initiates in the joint region at the joint-composite interface. Small amounts of porosity have been observed in certain areas of the joints. In addition to microstructural inhomogeneities in the joint regions, the mechanical strength of the joints is greatly influenced by delamination and failure at the fiber-matrix interface within the composite, which is weak by design. An example of the delamination and interface failure is shown in Fig. 6, which is the fractograph of a specimen tested at 1200°C.

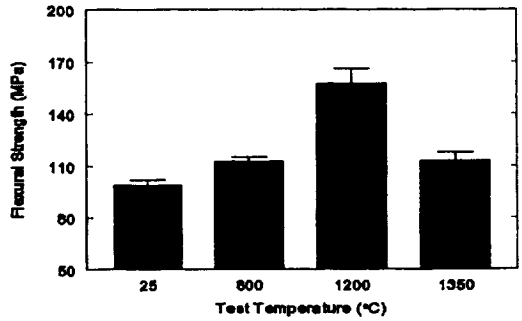


Fig. 5: Flexural strength of joined MI SiC/SiC composites at different temperatures.

The micrograph in Fig. 6 shows where one side of the joint pulled away from the weak fiber-matrix interface. This problem can be alleviated by using 3-D architectures or designing the joints in lap, scarf, or other configurations. Efforts are underway to fabricate joints with more homogeneous microstructure and composition, vary the joint configuration and thickness, and evaluate the effect of silicon-alloy infiltrants on the microstructure and mechanical properties of joints.

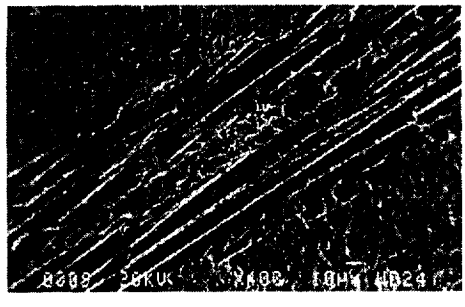


Fig. 6: SEM fractograph of joined MI SiC/SiC Composites tested at 1200 °C.

Conclusions

It has been demonstrated that the ARCJoinT approach can be used to join silicon carbide-based ceramics and fiber reinforced composites. These joints maintain their strength up to 1350°C in air. The strength of joints in RB-SiC is not limited by the joint strength, but by the strength of the bulk (parent) materials. Due to the low silicon content and uniform microstructure of the joints, the SiC grains cannot move freely without undergoing some deformation, resulting in high joint strength comparable to the strength of RB-SiC. However, in composites, low interfacial shear strengths act as the weak link and lead to low strength during flexure tests. Single or double lap configurations combined with different fiber architectures have to be explored for joints in composites.

ACKNOWLEDGMENTS

Author would like to thank Mr. R.F. Dacek for his help in the experimental work, Prof. Julian Martinez-Fernandez for compressive strength measurements, and to Mr. J.D. Kiser for critically reading the manuscript.

REFERENCES

1. Messler, Jr., R.W: Joining of Advanced Materials, Butterworth-Heinemann, Boston, MA (1993).
2. Schwartz, M.M.: Joining of Composite Matrix Materials, ASM International, Materials Park, OH (1994).
3. Fragomeni, J.M. and El-Rahaiby, S.K.: Review of Ceramic Joining Technology, Rept. No. 9, Ceramic Information Analysis Center, Purdue University, Indiana (1995).
4. Krenkel, W.; Henke, T.; and Mason, N.: Key Engineering Materials, 127-131 (1997) 313-320.
5. ASM Handbook, Materials Selection and Design, Vol. 20, ASM International, Materials Park, OH (1997).
6. Diefendorf, R.J.: Design with Composites, in Materials Selection and Design, ASM Handbook, (1997) Vol. 20, 648-665, ASM International, Materials Park, OH.
7. Singh, M.; Farmer, S.C.; and Kiser, J.D.: Ceram. Engg. and Sci. Proc., 18, 3 (1997) 161-166.
8. Singh, M. and Kiser, J.D.: in Physics & Process Modeling and Propulsion R&T Conference, NASA CP-10193, (1997) 5:1-10.
9. Singh, M.: Scripta Materialia, 34, 8 (1997) 1151-1154.
10. Singh, M.: Industrial Heating 9 (1997) 91-93.
11. Singh, M.: J. Mater. Sci. Letters, 17, 6 (1998) 459-461.
12. Singh, M.: J. Mater. Sci. 33 (1998) 5781-5787.
13. Singh, M.: Advanced Materials and Processes, 10 (1998) 89-90.
14. Singh, M.: Key Engineering Materials, 164 (1999) 415-420.
15. Martínez Fernández, J.; Muñoz, A.; Valera-Feria, F.M.; and Singh, M.: J. Europ. Ceram. Soc., 20 (2000) 2641-2648.
16. Martínez Fernández, J.; Muñoz, A.; Valera-Feria, F.M.; and Singh, M.: Joining of Advanced and Specialty Materials II, ASM International, Materials Park, OH, (2000) 25-33.
17. Singh, M. and Lara-Curzio, E.: ASME International, IGTE Turbo EXPO, Munich, Germany, paper no. 2000-GT-69 (2000) 1-6.